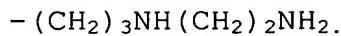


**Partially quaternized amino-functional
organopolysiloxanes and their use in aqueous systems**

5 This invention concerns partially quaternized amino-functional organopolysiloxanes and use thereof in aqueous system on textile substrates.

10 Organopolysiloxanes containing amino groups have long been used as textile finishes. Good and soft hand (hereinafter called softness effects) is achieved on textiles treated therewith. Preference is given to using organopolysiloxanes having the amino-functional group

15



Such amino-containing organopolysiloxanes when used in textile finishing are customarily present in the form 20 of aqueous microemulsions whose preparation is described in WO 88/08436 for example. Clear micro-emulsions are produced by heating a mixture of acid-neutralized amino-containing organopolysiloxane, water and emulsifier. According to the process described in 25 EP-A 0138 192, this goal is achieved by shear emulsification in concentrated form, containing little water, by proceeding from the basic ingredients of amino-containing organopolysiloxane in neutralized form, emulsifier and water.

30

Advantageous softness effects are obtained with 35 organopolysiloxanes whose amino functions are present in the form of the aminoethylaminopropyl groups mentioned. The morphological molecular structure of this side chain is believed to possess particular affinity for the fibre molecules of the textile substrate by embracing them. This results in the polymer molecule having a specific orientation which is responsible for the good softness. This is indirectly

corroborated by the fact that acylation (acetylation) of the outer, primary amino functions has an appreciable adverse effect on the softness of the textile substrates treated therewith, since the
5 different kind of molecular structure no longer permits adequate embracing of the fibre molecules and the associated specific orientation.

The amino-containing organopolysiloxanes described and
10 the organopolysiloxanes containing acylated amino groups are typically present as readily dissociable ammonium salts of organic or inorganic acids when in the form of their microemulsions. At above pH 7, the salts are converted into free bases. In the process,
15 the stability of the microemulsions is reduced by the weaker dissociation in the alkaline region, and this in the case of insufficiently acidified textile material and resultant pH values above 7 leads to coalescence of the microemulsion particles and their floating as oil
20 droplets on the surface of the treatment liquor. As the finishing process continues, the oil droplets can transfer to the textile material or else deposit on the rolls of the treatment assemblies. The textiles become stained with silicone spots which are very difficult or
25 impossible to wash off.

This behaviour in textile finishing is an appreciable disadvantage since very many treatment steps to finish textile substrates are carried out in a strongly
30 alkaline medium. Washing operations carried out with insufficient care may leave residual quantities of alkali on the substrate which are carried into the treatment baths during the subsequent finishing steps. Especially in equipment where the amount of treatment
35 liquor is not very large, as in the case of a pad for example, the pH may in the process very quickly rise to above 9 and lead to the split emulsions mentioned.

A further disadvantage of the amino-functional polysiloxanes identified is a tendency to yellow which can arise at drying temperatures above 120°C in the case of white and light-coloured textile substrates
5 treated therewith.

The amino-functional polysiloxanes identified are used on all textile substrates in the prior art. Woven and knitted fabrics composed of natural fibres, such as
10 cotton or wool for example, and also of synthetic fibres, such as viscose, polyester, polyamide or polyacrylonitrile for example, are successfully treated with such products on a large scale.

15 In some cases, it is a rather minor aspect of the finishing effects sought that amino-functional polysiloxanes endow textiles not only with good softness, but simultaneously also with a more or less pronounced, hydrophobic character. However, there are
20 textile applications where hydrophobicity is unwelcome. For instance, towels are expected to have not only a good, fleecy softness but also excellent absorbency to achieve an optimal drying effect. Good absorbency is frequently likewise desired for underwear. Similarly,
25 there are many clothing articles in the sports and outdoors sector, such as bicycling or soccer jerseys for example, where absorbency is a prerequisite for the textile base material as well as good softness. The use of amino-functional polysiloxanes is therefore limited
30 or completely impossible in the cases mentioned.

It is also known that microemulsions of organopolysiloxanes bearing quaternary ammonium groups do not have the disadvantages which have been described
35 with regard to thermal yellowing and with regard to stability in alkaline aqueous dilutions. As described hereinbelow, in some cases it is possible, depending on the method of making used and the composition, to achieve good hydrophilicity combined with good

absorbency. Organopolysiloxanes bearing quaternary ammonium groups consequently constitute an improvement over amino-containing organopolysiloxanes with regard to these identified properties. However, the identified
5 advantages of polysiloxanes bearing quaternary ammonium groups have to be weighed against the disadvantage that they, compared with polysiloxanes modified with lateral amino groups exclusively, generate less softness on textiles finished therewith.

10

Organopolysiloxanes bearing quaternary ammonium groups are known from the literature. Different ways are described to make them, and the quaternary ammonium groups of polysiloxanes obtained can be attached to
15 different positions on the polymer backbone of the polysiloxan chain depending on the particular starting materials used and the methods of making. Lateral, terminal and polymer backbone internal positionings of the functional groups and also mixed forms thereof are
20 possible.

For instance, DE-AS-14 93 384 describes a process for preparing organosiloxane compounds or compound mixtures wherein the corresponding methylhydrogenpolysiloxanes
25 are used as starting materials. The methylhydrogen-polysiloxanes in question are generally equilibrated siloxane mixtures in which the number of methylhydrogensiloxy and dimethylsiloxy units conform to a random distribution. The organosiloxanes having
30 quaternary ammonium groups are prepared in a conventional manner by reacting an epoxysiloxane with dimethylamine and converting the resulting dimethylaminoorganosiloxane with a hydrogen halide or with a methyl halide into the quaternary ammonium compound. The compounds prepared in this way are modified organopolysiloxanes where the quaternary ammonium groups are positioned laterally on the
35 polysiloxan chain. The compounds mentioned are recommended for the water-repellent treatment of, for

example, glass or aluminium surfaces.

A further way to prepare organopolysiloxanes having lateral quaternary ammonium groups is described in DE
5 19 652 524-A1. It involves, for example, aminoethylaminopropyl-containing organopolysiloxanes being reacted with methyl p-toluenesulphonate alkylating agent in the presence of water and suitable emulsifiers to form the corresponding quaternary
10 organopolysiloxanes, a microemulsion forming at the same time. The disadvantage of these preparations is the fact that the softness of textiles finished therewith is less pronounced than that obtained with polysiloxanes modified exclusively with lateral amino
15 groups.

The preparation and use of diquaternary polysiloxanes are described in US 4 891 166. The synthesis is effected by reacting polysiloxanes containing terminal
20 epoxy groups with tertiary amines in such ratios that there is at least one tertiary amino group for every · epoxy group and the reaction takes place in the presence of an acid equivalent, based on the nitrogen atoms to be quaternized, at elevated temperature. The
25 quaternary ammonium groups on the resulting diquaternary polysiloxanes are by virtue of this special method of preparation exclusively terminal-positioned. The compounds thus prepared are recommended for use in hair treatment agents and cosmetics. The
30 disadvantage of these preparations, as the Applicant has found, is the fact that textiles treated therewith have a less pronounced softness compared with polysiloxanes modified with exclusively lateral amino groups.

35

A further way to prepare quaternarily modified organopolysiloxanes is described in DE 37 05 121-A1. In this case, the method of preparation is such that the quaternary ammonium groups are exclusively positioned

within the polymer main chain. The method of polymerization involves for example a platinum-catalysed addition of allyl glycidyl ether onto α,ω -hydrogendimethylpolysiloxane and subsequent reaction of 5 the resulting α,ω -diepoxypolysiloxane with a ditertiary alkyldiamine under acidic conditions. Hair care is contemplated as a possible use. Again, the disadvantage of these preparations, as the Applicant has found, is the fact that textiles treated therewith have a less 10 pronounced softness compared with polysiloxanes modified with exclusively lateral amino groups.

WO 02/10259-A1 describes quaternarily modified organopolysiloxanes where the quaternary ammonium 15 groups are likewise positioned within the polymer main chain and have terminal, tertiary amino functions. They are recommended for textile finishing and also for cosmetic formulations.

20 Further polyquaternary polysiloxane polymers having quaternary groups disposed within the polymer main chain are described in US 4 533 714. They are used in cosmetic formulations for hair treatment.

25 US 3 207 707 describes the preparation of polymers having nitrogen in the main chain, the nitrogen being present in the form of tertiary amino groups, although no quaternary ammonium groups are formed. The products thus prepared are described inter alia for use as 30 packaging and insulating material.

US 3 033 815 describes the preparation of organopolysiloxanes having laterally disposed amino-functional groups and recommended for use as a size for 35 treating fibres, especially glass fibres.

WO 02/10501-A1 describes organopolysiloxanes for use as softeners having reduced foaming tendency which bear laterally disposed alkylated amino-functional groups.

US 5 039 738 provides another way to prepare organopolysiloxanes having laterally disposed tertiary amino-functional groups. They are said to provide 5 improved resistance to yellowing when used on textile substrates.

FR 1 184 198 A describes organofunctional alkylamino-alkylsilanes and organopolysiloxanes prepared therefrom 10 whose laterally disposed amino-functional groups are present in tertiary form.

Silanes and siloxanes having a terminal quaternary ammonium group are described in GB 1 006 729 as useful 15 for treating glass surfaces.

Cationic organopolysiloxanes are prepared in JP 02/157285-A. Because of only one terminal quaternary group, they have surface-active properties and are used 20 as surfactants.

Cationic siloxane copolymers bearing quaternary ammonium groups in the main chain are described in Journal of Polymer Science: Part A: Polymer Chemistry, 25 Vol. 40, 3570-3578 (2002). They are highly branched and are recommended for use as ion exchangers or in the sanitary sector.

The present invention then has for its object to 30 provide a preparation for treating textile substrates from aqueous media which on the one hand possesses the advantages of quaternary amino-functional organopolysiloxanes, such as hydrophilicity, stability at high pH values and yellowing resistance, while on 35 the other providing the good softness effects of organopolysiloxanes modified laterally with non-alkylated amino-functional groups.

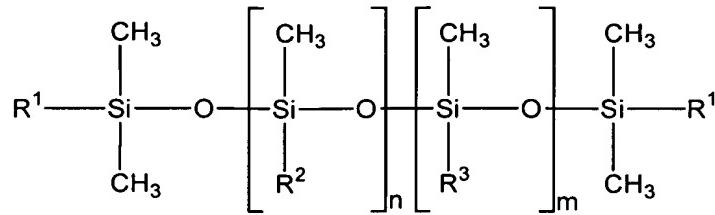
It has been determined that, unexpectedly, this object

is achieved according to the invention by the use of partially quaternized amino-functional organopolysiloxanes. Positioning of the quaternary ammonium groups on the polysiloxanes backbone is terminally in
5 the α,ω position and that of the amino-functional groups is laterally. Good stability under application conditions in a wide pH value range (especially between pH 7 and 12) and also high yellowing resistance are obtained. On the other hand, the preparations according
10 to the invention provide excellent softness. It has been determined that, surprisingly, the resulting softness is even distinctly superior to that of polysiloxanes bearing exclusively the terminal quaternary ammonium groups. Furthermore, textiles
15 finished with the preparations of the invention, especially towels, underwear and sportswear, are notable for high absorbency.

When the preparations according to the invention are
20 applied in the form of microemulsions, internal softness is preferentially obtained on cellulosic substrates since, owing to the low emulsion particle size of below 50 nm, the partially quaternized amino-functional polysiloxanes compounds are able to
25 penetrate deeply into the yarn and fibre interior. When preparations are used in the form of macroemulsions having particle sizes above 50 nm, deposition is preferentially achieved in the outer fibre and thread layers due to filtration effects. This leads to a
30 different hand character, featuring greater surficial smoothness and more fullness.

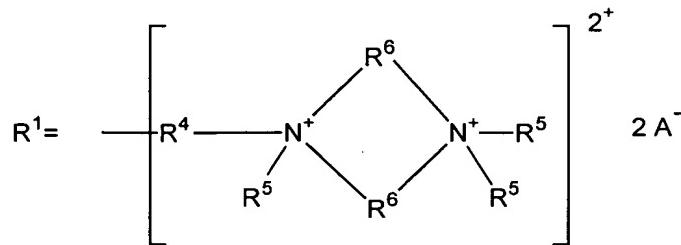
The present invention firstly provides preparations characterized by a content, based on the overall
35 composition, of

- (1) 2 to 60 per cent by weight of a partially quaternized amino-functional organopolysiloxane of the general formula



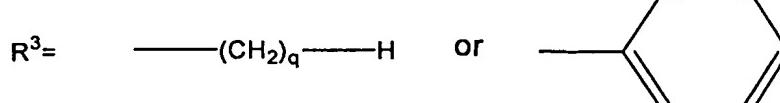
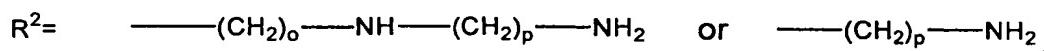
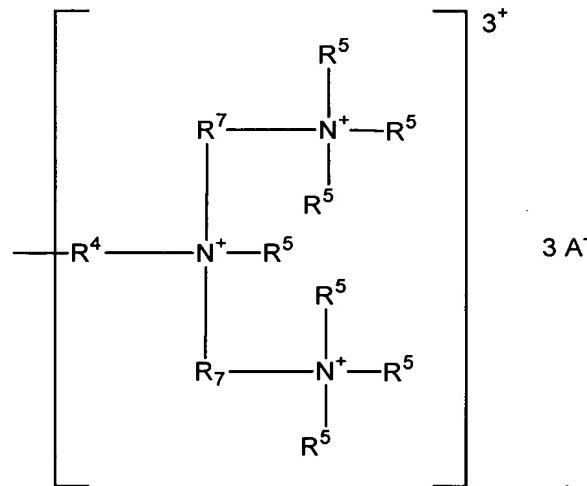
5

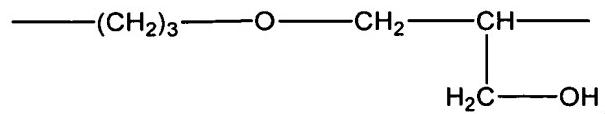
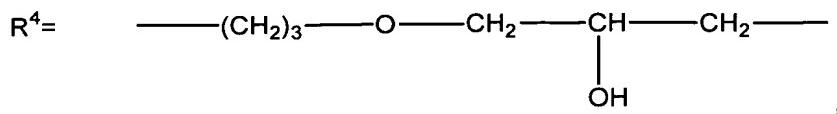
where



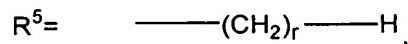
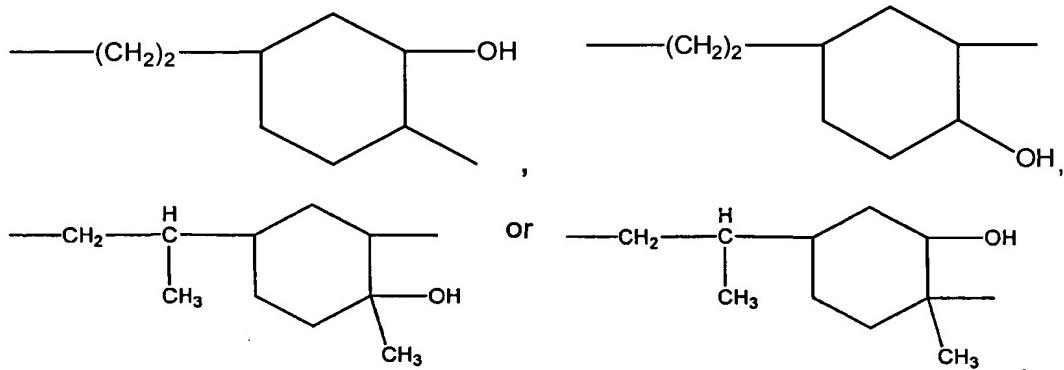
10

or

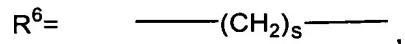




5



10



- A⁻ represents inorganic or organic anions,
n represents an integer from 1 - 20,
preferably 1 - 14 and more preferably 1 - 5,
m represents an integer from 20 - 2000,
preferably 40 - 1000 and more preferably
40 - 120,
o and p each represent an integer from 1 - 10
and preferably from 2 - 4,
q represents an integer from 1 - 10 and
preferably 1,
r represents an integer from 1 - 18 and
preferably 1,
s represents an integer from 2 - 3 and
preferably 2, and
t represents an integer from 2 - 5 and

preferably 2 - 4,

5 with the proviso that the total nitrogen content of component (1) is in the range from 0.05 to 2.0 per cent by weight, based on the overall composition of component (1),

- 10 (2) 2 to 40 per cent by weight of an emulsifier,
(3) 0 to 15 per cent by weight of a hydrotrope,
and
(4) 20 to 95 per cent by weight of water.

All the weight data for the preparations of the present invention are based on the overall composition of the preparation according to the present invention.
15 Preferred ranges are a range of 10 - 40 per cent by weight for component (1) and a range of 10 - 20 per cent by weight for component (2). When component (3) is added, its concentration is preferably in the range of 20 1 - 10 and especially of 3 - 7 per cent by weight. The preferred range for component (4) is 70-90 but especially between 60 - 90 per cent by weight.

The nitrogen content of component (1) is preferably 0.1
25 - 1.5 per cent by weight, but especially 0.1 - 1.0 per cent by weight based on the overall composition of component (1).

30 The anions are derived from inorganic or organic acids. Examples of inorganic anions include chloride, bromide, iodide and sulphate; chloride and sulphate are preferred. Examples of organic anions are tosylate and acetate; tosylate is preferred.

35 The partially quaternized amino-functional organopolysiloxanes of component (1) are prepared by methods known to one skilled in the art. Thus, the present invention's components (1) can be synthesized by equilibration of terminally quaternized amino-

functional organopolysiloxanes (preparable from terminally epoxy-functional organopolysiloxanes) with known silane hydrolysates bearing lateral amino groups in the presence of known equilibration catalysts.

5

Processes for analogous preparation of the terminally epoxy-functional organopolysiloxanes used as a starting compound are described for example in DE 3705121-A1, those for preparing the amino-functional intermediates 10 obtained therefrom for example in WO 02/10256-A1 and those for preparing the quaternary intermediates obtained therefrom in turn for example in DE 19652524-A1. The preparation of the known silane hydrolysates used is described for example by W. Noll in "Chemie und 15 Technologie der Silicone", 2nd edition 1968, page 168 et seq., and by M.A. Brook in "Silicon in Organic, Organometallic, and Polymer Chemistry", 2000, page 258 et seq.; aminoethylaminopropylsilane hydrolysates are commercially available. Known equilibration catalysts 20 include for example boron trifluoride, trifluoromethanesulphonic acid and sulphuric acid.

Useful emulsifiers (component (2)) include those on an anionic, cationic, nonionic or amphoteric basis or 25 mixtures thereof. Preference is given to using ethoxylation products of aliphatic alcohols having 6 to 22 carbon atoms which contain up to 50 mol of ethylene oxide in adducted form. The alcohols may preferably contain 8 to 16 carbon atoms; they can be saturated, 30 linear or preferably branched and can be used alone or mixed.

Of particular advantage with regard to low foaming in the application liquors are alcohols of the composition 35 mentioned when their alkylene oxide radical is constructed from ethylene oxide and 1,2-propylene oxide in random distribution and preferably in block distribution.

Nonionic emulsifiers from the group of ethoxylated branched aliphatic alcohols will be particularly advantageous because of their favourable overall properties. The preparations according to the invention
5 are therefore preferably made using for example ethoxylates of 2,6,8-trimethyl-4-nonal, of isodecyl alcohol or of isotridecyl alcohol each with 2 to 50 molecules and especially 3 to 15 molecules of adducted ethylene oxide.

10

The optional used component (3), a hydrotrope, can typically be selected from the group of polyfunctional alcohols. It is thus possible to use dialcohols having 2 - 10, preferably 2 - 6 but especially 2 - 4 carbon
15 atoms per molecule. Also of superior suitability are their mono- and diethers and also the mono- and diesters of these dialcohols. Examples of component (3) which are to be used with particular preference are butyldiglycol, 1,2-propylene glycol and dipropylene
20 glycol.

To bring the partially quaternized amino-functional organopolysiloxanes of Claim 1 into a form which can be applied from an aqueous medium, macro- or micro-
25 emulsions can be produced therefrom by high shear stirring of the components (1) to (4) at temperatures between 20 and 70°C.

The invention further provides for the use of the preparations according to Claim 1 in the finishing of textile substrates in aqueous baths and application liquors, if appropriate together with further preparation additives. Fabrics woven and knitted from natural fibres, such as cotton or wool for example, but
35 also from synthetic fibres, such as viscose, polyester, polyamide or polyacrylonitrile for example, can be treated with the amino-functional polysiloxanes of the invention. The preparation additives which can be used in addition, if appropriate, can be for example

chemicals for an wrinklefree finish, chemicals to improve the sewability of the textile substrate or other products typically used in textile application liquors.

5

The concentration of the preparations according to the invention in the application liquors is so chosen that the treated substrates contain between 0.3 and 0.6 per cent by weight of the partially quaternized amino-functional organopolysiloxanes of Claim 1, based on the weight of the substrate.

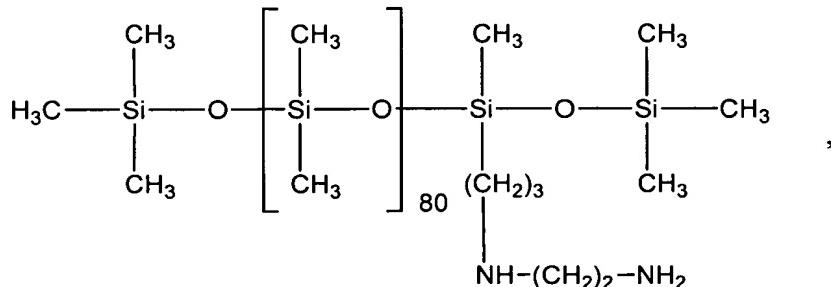
A preferred way to apply the preparations of the invention is as a forced application, by drenching the substrate with the application liquor, then squeezing it off on a padder and finally subjecting it to a drying passage.

The examples which follow illustrate the invention. The chain lengths reported in the formulae of these examples for indices n and m defined in Claim 1 are each an average value. The preparations used in the examples are produced by simply stirring the individual components together at 70°C.

25

Example 1 (not inventive):

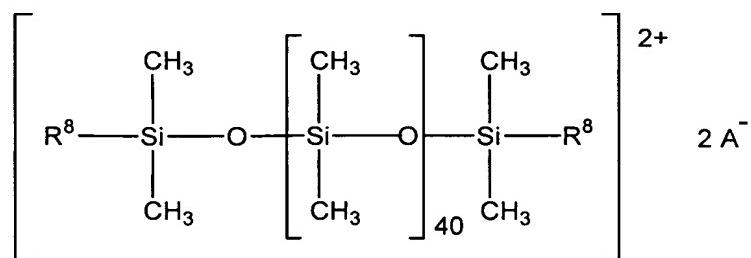
The preparation consists of an aqueous microemulsion containing 20 per cent by weight of an organopolysiloxane having laterally positioned amino groups of the formula



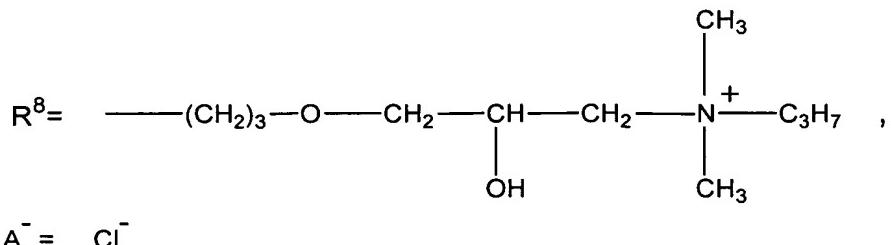
10 per cent by weight of an isodecyl alcohol with 7 ethylene oxide groups, 3 per cent by weight of butydiglycol and 0.4 per cent by weight of 60% acetic acid. The overall nitrogen content of the
5 organopolysiloxane is 0.44 per cent by weight.

Example 2 (not inventive):

The preparation consists of an aqueous microemulsion containing 20 per cent by weight of an
10 organopolysiloxane having terminally positioned and quaternized amino groups of the formula



15 where



20

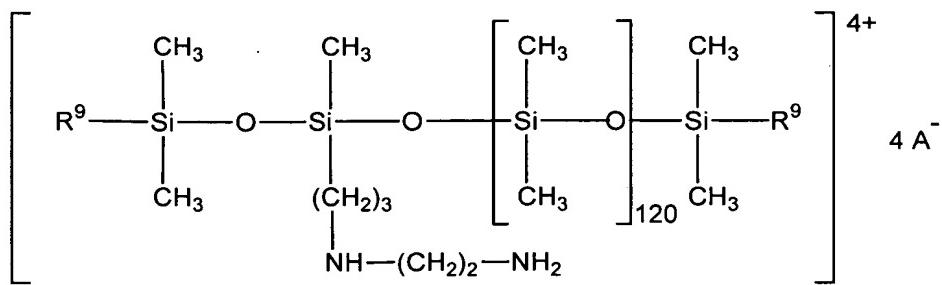
5 per cent by weight of an isotridecyl alcohol with 6 ethylene oxide groups, 5 per cent by weight of an isodecyl alcohol with 7 ethylene oxide groups and 7 per cent by weight of butydiglycol. The overall nitrogen
25 content of the organopolysiloxane is 0.78 per cent by weight.

Example 3 (inventive):

The preparation consists of an aqueous microemulsion
30 containing 20 per cent by weight of an

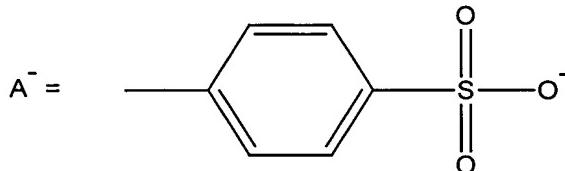
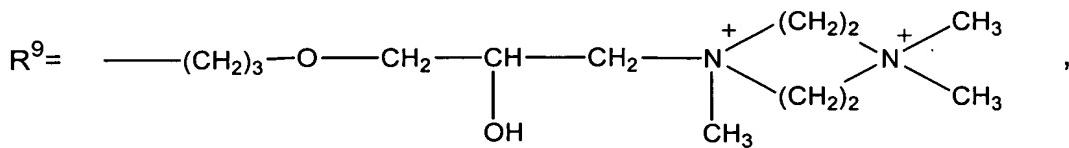
organopolysiloxane having laterally positioned amino groups and also terminally positioned and quaternized amino groups of the formula (I)

5



where

10



5 per cent by weight of an isotridecyl alcohol with 6 ethylene oxide groups, 5 per cent by weight of an 15 isodecyl alcohol with 7 ethylene oxide groups and 7 per cent by weight of butyldiglycol. The overall nitrogen content of the organopolysiloxane is 0.80 per cent by weight.

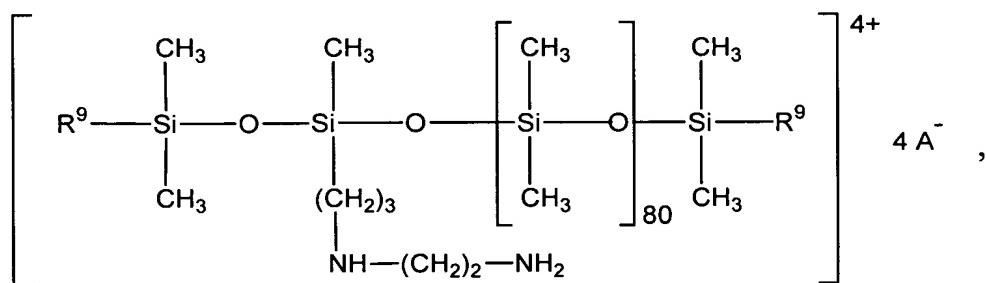
20 The organopolysiloxane of the formula (I) is prepared in a similar manner to Part 1a) of Example 1 of WO 02/=10256-A1 by using a polydimethylsiloxane having terminal epoxy groups and an average chain length of 120 units, prepared in a similar manner to Example 1 of 25 DE 3705121-A1. The intermediate product obtained is quaternized with methyl p-toluenesulphonate in isopropanolic solution in a manner similar to DE 19652524-A1 and, after the solvent has been distilled

away, admixed with 2 per cent by weight of an aminoethylaminopropylsilane hydrolysate. Following the addition of 0.15 per cent by weight of trifluoromethanesulphonic acid the batch is stirred at 5 115°C for 2 hours during which time the originally inhomogeneous, cloudy mixture converts into a clear, yellowish organopolysiloxane of the formula (I).

Example 4 (inventive):

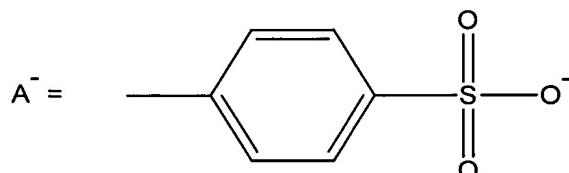
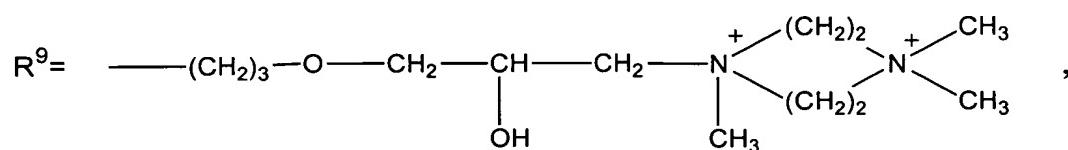
10 The preparation consists of an aqueous microemulsion containing 20 per cent by weight of an organopolysiloxane having laterally positioned amino groups and also terminally positioned and quaternized amino groups of the formula (II)

15



where

20



5 per cent by weight of an isotridecyl alcohol with 6 25 ethylene oxide groups, 5 per cent by weight of an isodecyl alcohol with 7 ethylene oxide groups and 7 per cent by weight of butyldiglycol. The overall nitrogen content of the organopolysiloxane is 1.13 per cent by

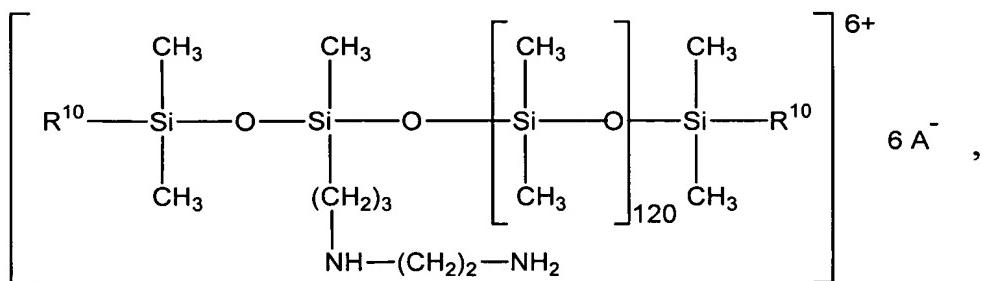
weight.

The organopolysiloxane of the formula (II) is prepared in a manner similar to Example 3 by using a polydimethylsiloxane having terminal epoxy groups and an average chain length of 80 units. A clear yellowish organopolysiloxane is formed.

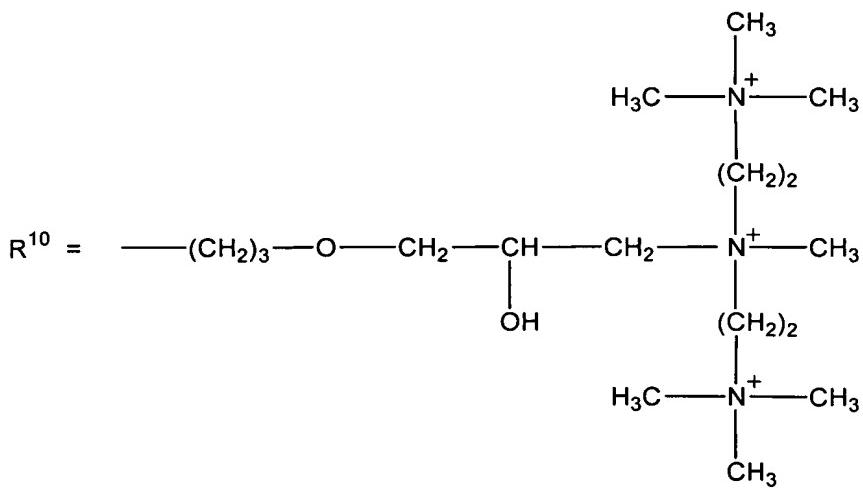
Example 5 (inventive):

10 The preparation consists of an aqueous microemulsion containing 20 per cent by weight of an organopolysiloxane having laterally positioned amino groups and also terminally positioned and quaternized amino groups of the formula (III)

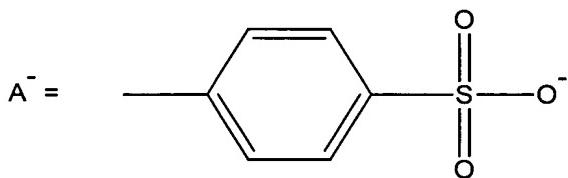
15



where



20



- 5 per cent by weight of an isotridecyl alcohol with 6 ethylene oxide groups, 5 per cent by weight of an
5 isodecyl alcohol with 7 ethylene oxide groups and 7 per cent by weight of butyldiglycol. The overall nitrogen content of the organopolysiloxane is 1.03 per cent by weight.
- 10 The organopolysiloxane of the formula (III) is prepared in a similar manner to Example 4 of WO 02/10501-A1 by using a polydimethylsiloxane having terminal epoxy groups and an average chain length of 120 units, prepared in a similar manner to Example 1 of DE
15 3705121-A1. The intermediate product obtained is quaternized with methyl p-toluenesulphonate in isopropanolic solution in a manner similar to DE 19652524-A1 and, after the solvent has been distilled away, admixed with 2 per cent by weight of an
20 aminoethylaminopropylsilane hydrolysate. Following the addition of 0.15 per cent by weight of trifluoromethanesulphonic acid the batch is stirred at 115°C for 2 hours during which time the originally inhomogeneous, cloudy mixture converts into a clear,
25 yellowish organopolysiloxane of the formula (III).

Use examples

30 **Hand assessment**

Portions cut from bleached cotton terry towelling which had not been optically brightened were impregnated with an aqueous liquor containing 20 g/l of the micro-emulsions produced according to the examples and
35 0.5 g/l of 60% acetic acid on a laboratory padder to a wet pick-up of 80% and then dried at 120°C for 2

minutes. The hand character of the test fabrics treated with the microemulsions was subsequently assessed. This assessment is subject to individually different, subjective criteria. To obtain meaningful results nonetheless, at least 5 judges have to do the assessing. The results were evaluated by statistical methods, a rating of 1 denoting the softest, most pleasant hand and a rating of 10 denoting the harshest, least superficially smooth and most unpleasant hand within the test series.

	Hand assessment rating within test series
Microemulsion of Example 1	1.5
Microemulsion of Example 2	7.2
Microemulsion of Example 3	2.1
Microemulsion of Example 4	2.6
Microemulsion of Example 5	3.4
Untreated	10

Hydrophilicity

Portions cut from bleached cotton terry towelling which had not been optically brightened were impregnated with an aqueous liquor containing 20 g/l of the micro-emulsions produced according to the examples and 0.5 g/l of 60% acetic acid on a laboratory padder to a wet pick-up of 80% and then dried at 120°C for 2 minutes. The hydrophilicity was subsequently assessed according to the TEGEWA drop test (Melliand Textilberichte 68 (1987), 581 - 583).

	Sink time (sec)
Microemulsion of Example 1	above 180
Microemulsion of Example 2	10
Microemulsion of Example 3	below 1
Microemulsion of Example 4	1
Microemulsion of Example 5	3
Untreated	below 1

Yellowing

Samples cut from bleached cotton-modal knit fabric which had not been optically brightened were
5 impregnated with an aqueous liquor containing 20 g/l of the microemulsions produced according to the examples and 0.5 g/l of 60% acetic acid on a laboratory padder to a wet pick-up of 80% and then dried at 120°C for 2 minutes and subsequently cured at 170°C for 2 minutes.
10 The whiteness of the samples was then measured after Ganz on a "texflash 2000" whiteness meter from "datacolor international" (Switzerland).

	Ganz whiteness
Microemulsion of Example 1	141.5
Microemulsion of Example 2	151.0
Microemulsion of Example 3	150.1
Microemulsion of Example 4	147.8
Microemulsion of Example 5	150.5
Untreated	151.8

15

Alkali stability

The stability of the organopolysiloxane emulsions to alkalis in finishing liquors was tested according to the hereinbelow described test:

20 500 ml of a solution of the in-test organopolysiloxane emulsion having a concentration of 40 g/l were placed into a 1000 ml glass beaker as initial charge and adjusted to pH 12 with sodium hydroxide solution (w(NaOH) = 10%). The liquor was subsequently stirred
25 with a blade stirrer at 2000 revolutions per minute for twenty minutes. Upon expiration of this time the stirrer was switched off, the foam which had formed was allowed to collapse and the liquid surface was inspected after 1 hour for the presence of floaters
30 (separated material which had floated to the surface of the liquid).

	Evaluation after 1 hour
Microemulsion of Example 1	substantial silicone floaters
Microemulsion of Example 2	thin oil film on surface
Microemulsion of Example 3	no floaters
Microemulsion of Example 4	no floaters
Microemulsion of Example 5	no floaters